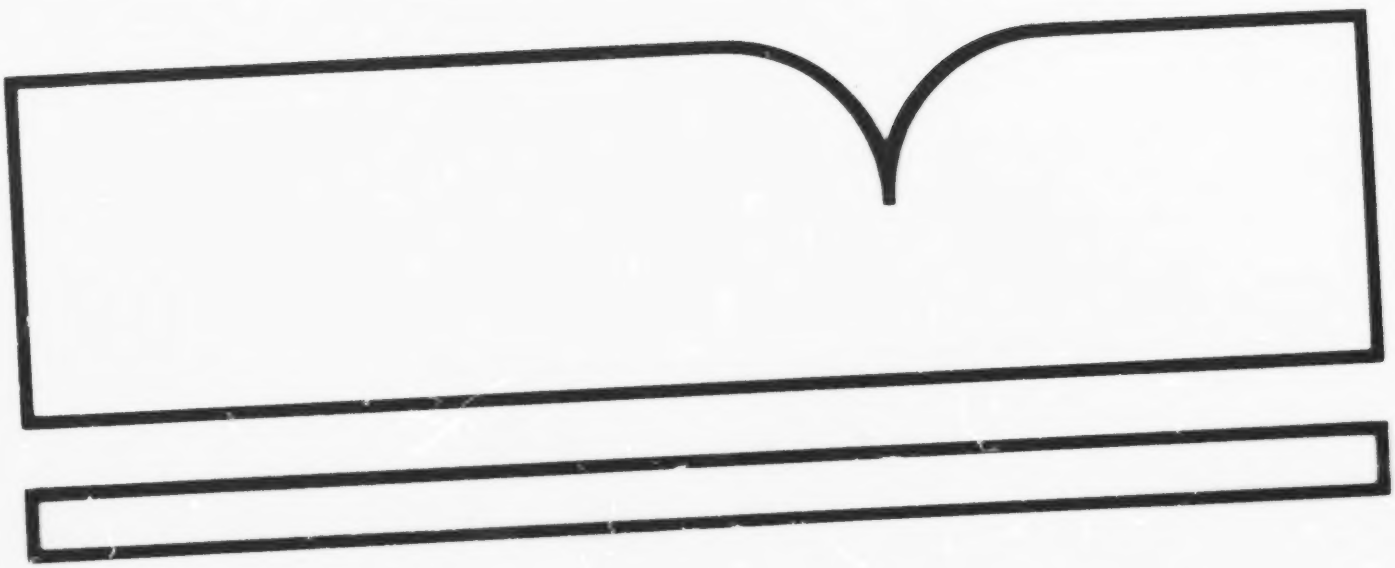
PB90-116757

Large Laboratory Column Study of the Transport and Degradation of Atrazine, Carbofuran, and Diuron in Soils

(U.S.) Robert S. Kerr Environmental Research Lab., Ada, OK

1989



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TECHNICAL REPORT DATA (Please read Instructions on the reverse before complet		
1. REPORT NO. EPA/600/D-89/162	2.	3. PB90-116757
4. TITLE AND SUBTITLE LARGE LABORATORY COLUMN STUDY OF THE TRANSPORT AND DEGRADATION OF ATRAZINE, CARBOFURAN, AND DIURON IN SOILS		5. REPORT DATE 6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Thomas E. Short and Carl G. Enfield		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Robert S. Kerr Environmental Research Laboratory U.S. Environmental Protection Agency P.O. Box 1198 Ada, OK 74820		10. PROGRAM ELEMENT NO. ABWD1A 11. CONTRACT/GRANT NO. In-House
12. SPONSORING AGENCY NAME AND ADDRESS Robert S. Kerr Environmental Research Laboratory U.S. Environmental Protection Agency P.O. Box 1198 Ada, OK 74820		13. TYPE OF REPORT AND PERIOD COVERED Symposium Paper 14. SPONSORING AGENCY CODE EPA/600/15

15. SUPPLEMENTARY NOTES IN: Methodological Aspects of the Study of Pesticide Behaviour in Soil, P. Jamet, Ed., Versailles, France, INSTITUT NATIONAL DE LA RECHERCHE AGRONOMIQUE, pp. 139-150, 1989.

16. ABSTRACT

The suitability for land treatment of 3 pesticides in a liquid waste stream was evaluated. The evaluation was performed under laboratory conditions utilizing 15 cm by 150 cm long glass columns. Composition of the water was monitored as a function of time and depth for eight columns utilized in the study. Experimental columns were duplicated and there were 4 treatments in the study. Three different soils were studied and one soil was studied at two different hydraulic loading rates. The experimental data were collected in both batch static microcosms and dynamic columns studies. The confidence limits on the batch obtained transformation rates were not sufficiently tight to permit resaonable projections in most cases. The pesticides followed a systematic pattern in the column with respect to transport and degradation. However, apparently processes other than first order take place in the profile and it is necessary to perform additional studies to delineate the actual biochemical processes.

17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group

REPRODUCED BY
U.S. DEPARTMENT OF COMMERCE
NATIONAL TECHNICAL INFORMATION SERVICE
SPRINGFIELD, VA. 22161

18. DISTRIBUTION STATEMENT	19. SECURITY CLASS (This Report)	21. NO. OF PAGES
RELEASE TO PUBLIC	UNCLASSIFIED	16
	20. SECURITY CLASS (This page) UNCLASSIFIED	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

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Editor / *Editeur*

P. JAMET
INRA, Station de Phytopharmacie
Rte de St Cyr, 78026 Versailles Cedex

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ISBN : 2-7380-0115-7

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EPA/600/D-89/162

Methodological aspects of the study of pesticide behaviour in soil

Aspects méthodologiques de l'étude du comportement des pesticides dans le sol

INRA Versailles, June 16-17, 1988

Edited by
Paul JAMET

INSTITUT NATIONAL DE LA RECHERCHE AGRONOMIQUE
147, rue de l'Université, 75341 Paris Cedex 07

Methodological aspects of the study of pesticide behaviour in soil. INRA Versailles, June 16-17, 1988.

LARGE LABORATORY COLUMN STUDY OF THE TRANSPORT AND DEGRADATION OF ATRAZINE, CARBOFURAN, AND DIURON IN SOILS

Thomas E. Short
Carl G. Enfield

Bureau de Recherches Géologiques et Minières
B.P. 6009 - 45060 ORLEANS cedex 2 (France)

Robert S. Kerr Environmental Research Laboratory
P.O. Box 1198 - Ada, Oklahoma 74820 (U.S.A.)

Summary

The suitability for land treatment of 3 pesticides in a liquid waste stream was evaluated. The evaluation was performed under laboratory conditions utilizing 15 cm by 150 cm long glass columns. Composition of the water was monitored as a function of time and depth for eight columns utilized in the study. Experimental columns were duplicated and there were 4 treatments in the study. Three different soils were studied and one soil was studied at two different hydraulic loading rates. The experimental data were collected in both batch static microcosms and dynamic columns studies. The confidence limits on the batch obtained transformation rates were not sufficiently tight to permit reasonable projections in most cases. The pesticides followed a systematic pattern in the column with respect to transport and degradation. However, apparently processes other than first order take place in the profile and it is necessary to perform additional studies to delineate the actual biochemical processes.
Key words: Transport, Sorption, Degradation, Atrazine, Carbofuran, Diuron, Soil.

Résumé

Trois pesticides ont été testés dans un cours d'eau usée pour déterminer s'ils peuvent convenir pour le traitement des sols. L'étude fut réalisée dans des conditions de laboratoire en utilisant de grands tubes de verre de 15 par 150 cm. La composition de l'eau utilisée dans les huit tubes de l'expérience a été choisie selon des périodes et des profondeurs différentes. Les tubes expérimentaux furent répétés et quatre traitements furent réalisés. Trois sols différents furent étudiés et un sol fut étudié en fonction de deux niveaux de remplissage hydrauliques différents. Les données de l'expérience furent recueillies à la fois sous une forme globale de microcosmes statiques ainsi que sous une forme d'études de tubes dynamiques. Les limites de fiabilité dans la forme globale obtinrent des taux de transformation qui ne s'avérèrent pas suffisamment légers pour permettre des projections acceptables dans la plupart des cas. Les pesticides ont présenté des caractéristiques systématiques dans le tube en fonction du transport et de la dégradation. Cependant, des processus apparemment différents des précédents apparaissent dans l'étude et il faut recourir à des études supplémentaires pour connaitre les processus biochimiques réels.
Mots-cles : Transport, Sorption, Dégradation, Atrazine, Carbofuran, Diuron, Sol.

Introduction

The principal objective of the present study was to evaluate the transport of pesticides and other hazardous organic chemicals through soils at land treatment sites. Land treatment is one of the techniques for anagement of hazardous wastes. In this treatment technique, the naturally occurring properties of the soil are used to convert the wastes to nonhazardous components. It is conceptualized that at the land treatment facility the soil will completely assimilate the applied waste. The physical, chemical and microbiological processes work in such a way that the waste constituents will not leach beyond the treatment zone. Thus the possibility of contaminating ground water will be ideally eliminated. Avoiding the accumulation of degradation by-products that may be detrimental to human health and the environment is also a concern. Wastes commonly disposed of at land treatment facilities are characterized as being a mixture of a wide variety of components. Most of these components are capable of being readily degraded by soil microorganisms. However, these wastes often contain small amounts of hazardous compounds that are more resistant to degradation.

The main objective of this study was to use soil columns (simulating land treatment) to gain more knowledge about the transport and transformation processes. In this study, two totally independent sets of experimental data were collected. Soil columns simulating land treatment systems were operated to collect dynamic data documenting the transport and transformation of contaminants in the experimental systems. Simultaneous batch (or static) experiments were performed in an attempt to obtain corresponding information.

The objective of this paper is to describe and discuss the operation of the dynamic column study and the methods and results of the static studies.

Soils

The three soils selected for this study were characterized by Nkedi-Kizza and Rao (1987). A summary of their characterization is presented in Table 1. Eustis soil from Florida, Lincoln soil from Oklahoma, and Kidman soil from Utah were utilized. Soil samples were collected from the top 30 cm of the soil profile, air-dried, and sieved to pass through a 2 mm sieve.

Table 1. Physical and Chemical Properties of Soils

Soil Series	Particle Size* Fraction %			Organic Carbon (%)	pH 1:2 Suspension (Water)	CEC (meg/100g Soil)
	Sand	Silt	Clay			
Eustis FS	91.6	4.2	4.2	0.78	5.8	9.60
Lincoln S	88.6	9.4	2.0	0.22	6.8	3.75
Kidman FSL	54.0	31.4	14.6	0.61	8.0	24.43

* Pipette Method

Soil Columns

Glass columns 15 cm I.D. X 150 cm long with sampling ports at 7.5, 15, 30 and 60 cm were constructed. A total of eight soil columns were packed with the three soils. Four columns were packed with Lincoln soil, 2 columns with Eustis soil and 2 columns with Kidman soil. The saturated hydraulic conductivities of the columns packed with Lincoln, Eustis, and Kidman soils were measured at 2.8 $\pm$ 0.2, 8.5 $\pm$ 0.2, and 0.6 $\pm$ 0.02 cm/hr,

respectively. The sampling ports were fitted with solution samplers composed of a cylinder of sintered glass (1 cm I.D. and 2 cm long) with maximum pore size of 4 to 8 μm. The samplers were positioned at the center line of the soil columns. The top of each column was enclosed in a "greenhouse" 45 cm high which was used to determine the rate of volatilization of the compounds. The columns were housed in a constant temperature room at 20 ± 2°C.

The waste stream was applied at the top of the soil columns with two pumps at two flow rates. Two Lincoln soil columns and the two Kidman soil columns received the waste stream at an average flow rate of 0.06 cm/hr. For the remaining 2 soil columns of Lincoln soil and the two Eustis soil columns, the waste stream was applied at a higher flow rate averaging 0.28 cm/hr. The flow rates in the columns were thus selected to be no more than 10% of the saturated hydraulic conductivity in any of the columns in order to achieve steady state unsaturated flow. The waste stream was applied to the soil columns for 1 minute for the slow flow rate and for 2 minutes for the fast flow rate at four-hour intervals. Figure 1 contains a schematic diagram of the columns used in this study.

Chemicals Selected for the Waste Stream

The composition of the synthetic waste stream is presented in Table 2. The waste stream consisted of 5 categories of material. The first category, the substrate, is highly degradable and is intended to serve as food for the microorganisms present in the soil. Sego^{R} the primary substrate material is a commercially available milk based dietary supplement for human consumption. The application of Sego^{R} was intended to enhance microbial growth and to provide a uniform microbial population in the soil columns. Methanol, another substrate material, is also degradable and acts as a food source for the microbes. However, the primary purpose of the methanol is to aid in the dissolving of the less soluble components of the waste stream. The pesticides were included as reference compounds because the environmental behavior (sorption, transport and degradation) of these chemicals has been characterized previously. The aromatic hydrocarbons and the phenols were selected because these types of chemicals are commonly contained in wastes that are land treated. Tritiated water was used as a reference non-adsorbed conservative solute in order to characterize the dynamics of water flow in the soil columns.

Table 2. Composition of the Waste Stream

Category	Name	Concentration(mg/l)
Substrate	Sego^{R}	7 (ml/19L)
Substrate	Methanol	10 (ml/19L)
Pesticide	Diuron	10
Pesticide	Atrazine	10
Pesticide	Carbofuran	10
Aromatic	Benzene	2.5
Aromatic	Toluene	2.5
Aromatic	Naphthalene	2.5
Aromatic	p-Xylene	1
Phenol	Phenol	10
Phenol	2,4-Dichlorophenol	10
Phenol	2,4-Dimethylphenol	10
Phenol	2,4-Dinitrophenol	10
Phenol	Pentachlorophenol	0.2
Tritiated Water	$^{3}H_2O$	10000 (dpm/ml)

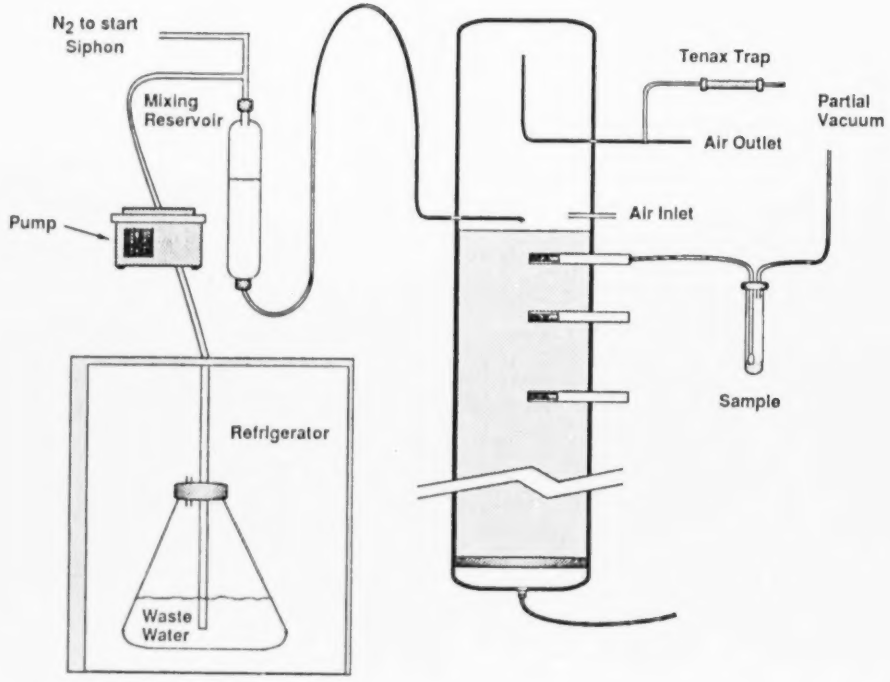


Figure 1. Schematic of Large Column Apparatus

Sego^{R} Application to Soil Columns

The soil columns received Sego^{R} at a rate of 7 ml per 19 L of filter-sterilized ground water for at least 3 months before the waste stream was applied to the soil columns at the two flow rates outlined earlier. The application of Sego^{R} was intended to enhance microbial growth and to provide a uniform microbial population in the soil columns. To avoid degradation of Sego^{R}, the feed solution was prepared every three days and was stored in a refrigerator.

Preparation of Waste Stream

The waste stream was prepared from three stock solutions of the organic chemicals.

Solution 1

The "volatile" cocktail contained benzene, toluene, naphthalene and p-xylene. These compounds were added step-wise to a 1000 ml volumetric flask containing methyl alcohol in this order: toluene, benzene, p-xylene and naphthalene. When made to volume, the stock solution was split into 5 ml increments in glass ampules and fire-sealed under nitrogen. Four ml of the stock was added to 19 L of filter-sterilized water. The final concentration of each chemical in the feed solution was 2.5, 2.5, 1 and 2.4 mg/L for toluene, benzene, p-xylene and naphthalene, respectively. These organic chemicals were analyzed by gas chromatography.

Solution 2

Solution 2 consisted of the "nonvolatile" compounds. These compounds were dissolved in methanol step-wise. To 1000 ml of methanol 10 g of 2,4-DMP, 10 g of 2,4-DCP, 10 g of phenol, 2.5 g of atrazine, 10 g of diuron and 10 g of carbofuran were added. The stock solution was split into 5 ml portions, sealed in glass ampules under nitrogen and stored in the dark at 4^{0}C. Table 2 shows the final concentration in the feed solution when 5 ml of the stock solution was added to 19 ml of water. These chemicals in solution 2 were analyzed by liquid chromatography.

Solution 3

Solution 3 consisted of pentachlorophenol (PCP). C-12 PCP was dissolved in methyl alcohol and then spiked with C-14 PCP to give final concentration in the feed solution of 0.2 ml/L of C-12 and about 2000 cpm/ml of C-14. Initially $^{3}H_{2}O$ was added to solution 3 such that when an aliquot was added to 19 L of water tritium counts were about 10,000 dpm/ml. Solution 3 was analyzed by liquid scintillation counting.

Sego^{R} was also added to the feed solution containing all 13 organic chemicals. About 7 ml of Sego^{R} was added to 19 L of water. Thus, the final feed solution contained all chemicals in Table 2 plus Sego^{R}. The 19 L feed solution was prepared every 3 days for about 7 months and was stored under constant refrigeration.

Maintenance of Soil Columns

Initially soil columns were wetted with filter-sterilized water on December 15, 1984. Sego^{R} at a rate of 7 ml per 19 L of water was applied from January 15, 1985. Sego^{R} along with all organic chemicals and tritium was applied on July 16, 1985. The application of chemicals to soil columns was halted on August 30, 1985 due to "poisoning" of soil microbes in columns suspected to be caused by 60 ml of methanol that was used to dissolve organic chemicals in each 19 L of water prepared every 3 days. Water was then applied to the soil columns beginning August 30, 1985 to leach methanol out of the soil columns. Sego^{R} solution was re-applied on November 6, 1985 to all columns. New feed solution was prepared every 3 days until the experiment was stopped in May-June 1986. Tritium was applied for only 3 days beginning on November 6, 1985. Solution samples were taken from 3 ports for each soil column except for two columns where

samples were taken at the bottom of the columns. Each port was sampled at least once a week. Effluent flow rates were measured twice a week for each column. The liquid samples from the column were analyzed for organic chemicals as outlined earlier. At the end of the experiment, soil columns were dismantled and soil samples were taken at every 15 cm depth. The moisture content of these subsamples was determined. These samples were also analyzed for organic chemicals content by repeated extractions using 90% methanol. Each sample (94 g soil plus 10 ml of solution) was extracted on a platform shaker for 3 hours and centrifuged for 30 minutes at 1200 rev/min (arm radius 150 mm, Sorvall RT 6000). The supernatant was then analyzed for each component.

Sampling of Soil Columns

Three types of samples were taken from the soil columns. The samples are designated here according to the method by which they were analyzed.

Liquid Chromatography and Liquid Scintillation Samples

The solutions from the sampling ports were obtained with a vacuum of about 30 kPa and collected in 20 ml vials. Samples at the bottom of the soil columns were taken without applying a suction. The samples were filtered through aqueous millipore (0.45μ) filters before being analyzed for the nonvolatile compounds.

Gas Chromatography Head Space Sampling

The head space samples were collected for a period of 24 hours. A Tenax trap was connected to the head space port and a vacuum of about 500 mm Hg was applied. Flow rate through the trap was measured at the beginning and at the end of the sampling period using a soap film flow meter.

Gas Chromatography Samples

For sampling the feed solution and the effluent at the bottom of the columns, a syringe with tubing attached was used to pull a 6 ml liquid sample. For all samples, the elapsed time during sampling was recorded.

Procedures for Sample Handling

Samples were appropriately identified according to soil series, depth in soil column, method of collection, and date and time of collection. Samples were usually analyzed on the day they were collected, otherwise they were refrigerated for no longer than two days before they were analyzed.

Liquid Chromatography Analysis of Phenols and Pesticides

A method was developed for the analysis of phenol, 2,4-dinitrophenol, 2,4-dimethylphenol, 2,4-dichlorophenol and the pesticides atrazine, diuron, and carbofuran. Due to the high polarity (phenols) and thermal instability (carbofuran) of some of the compounds, reversed-phase high performance liquid chromatography was the analysis method of choice.

The classical technique of base extraction for separation of weak acids (phenols) from neutral mixtures was not applicable in this study due to carbofuran's instability in base. Low sample volume (0.4-4.0 ml) precluded a practical concentration step. Autosampler precision and UV detector stability made external standard quantitation the method of choice.

A small volume (0.4-4.0 ml) of sample from the column was filtered through a 0.45 μ filter. The filtrate was collected in an autosampler vial and injected on the liquid chromatograph (10-100 μl injection volume) without further sample clean-up. The limit of detection for all analytes was better than 0.1 ppm using this injection technique.

The mobile phase consisted of 41% methanol and 7% acetonitrile. The aqueous phase was prepared by acidifying water to pH=2 with hydrochloric acid before combining with the mobile phase. All mobile phase solvents were degassed before use by filtering through a 0.45 μ filter under vacuum. Flow rate was 1.6 ml/min and the analytical column was a C-18 Nova-pak (Water's Assoc., approximately 6500 plates). Standards of each analyte were prepared and scanned to determine analytical wavelength and to acquire a library of scans for identification of analytes. Scans of actual samples were performed periodically to corroborate tentative identification of analytes by retention times.

Each analysis session began by calibrating the instrument with a standard solution of the analytes or by comparing the calculated concentrations to the previous session's calibration. The instrument was satisfactorily calibrated if the measured values were within 5% of the true values. Controls (Column eluate without analytes) were run before the inception of the experiment, but were not available once the experiment was in progress.

Effluent Breakthrough Curves for Pesticides

The scope of this paper does not permit a full presentation of all breakthrough curves that were obtained in this study. However, we can present some examples of the types of outputs that were observed. Figure 2 contains the breakthrough curve for carbofuran from the bottom of column A. This breakthrough curve behaves as would be expected for a compound that was retarded due to sorption and degrades only a small amount. The column reached steady state rapidly.

Figure 3 contains the breakthrough curve for atrazine from the bottom of column B. This breakthrough curve also shows retardation due to sorption. However, the curve does not reach a steady state despite the fact that all the runs were conducted over a nine-month period. It appears as though the microbes in the column are slowly being acclimated to the feed; and, as time goes on, their ability to degrade atrazine improves.

Figure 4 contains the breakthrough curve for carbofuran from the bottom of column F. Again, the curve shows the effect of retardation due to sorption. It also appears to reach steady state rapidly. However, at about 11 pore volumes there is a sudden drop in the output concentration of the carbofuran. It is as though there was a rapid change in the ability of the microbes to degrade the carbofuran. This could possibly be due to a shift in the microbial population within the column.

Estimation of Half-Life from Breakthrough Curves

The half-life values for the pesticides in the three soils calculated from breakthrough curves are presented in Table 3. The half-lives were calculated by using the known pore water velocity, the depth of the port, and the observed attenuation factor for the concentration of the pesticide at steady state. When steady state concentrations were not reached, the half-life could not be calculated.

In Table 4, half-life values obtained from the batch method (static system) are presented. These data were furnished by Dr. Sims, Utah State University. These half-life values have a wide confidence interval, and most fall within the half-life values obtained in this study.

Sorption

Sorption of organic pollutants plays a significant role in determining their fate and transformation in the environment. A considerable effort has been expended to determine sorption coefficients for single sorbates in soils. However, from a practical approach, multiple organic sorbates are expected in complex systems commonly encountered at hazardous waste land treatment sites. This necessitates determination of sorption from both single-sorbate and multi-sorbate systems to determine if

Figure 2. Carbofuran, Col. A

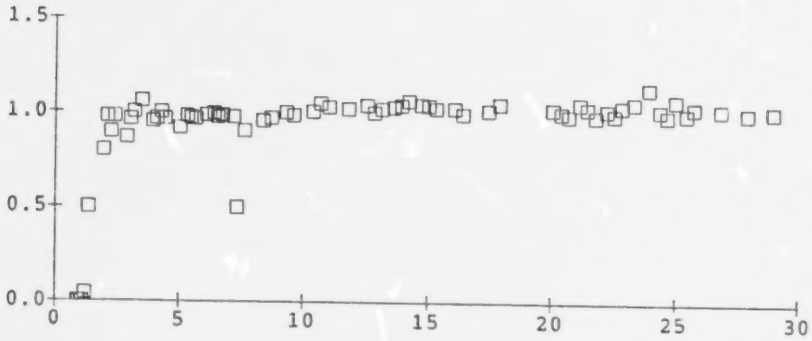


Figure 3. Atrazine, Col. B

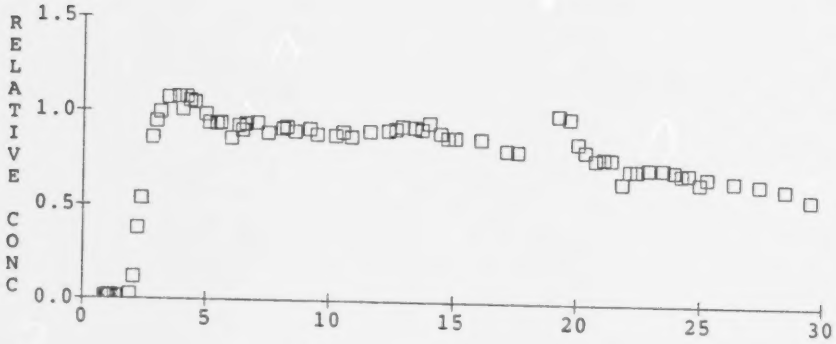
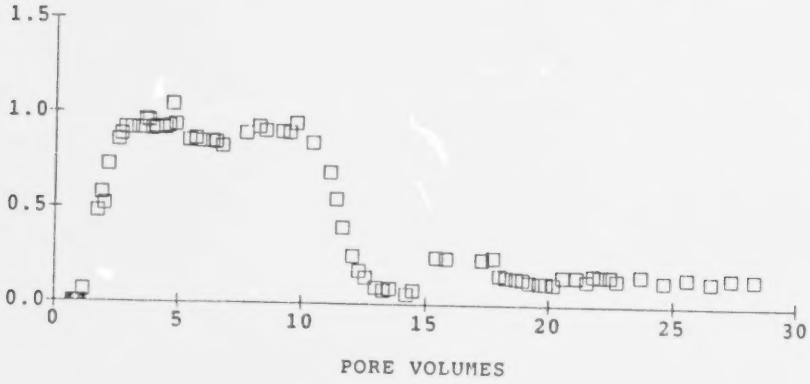


Figure 4. Carbofuran, Col. F



there is any effect of competitive sorption due to the presence of other organic chemicals in the mixture.

Table 3. Calculated Half-Lives (days) from Breakthrough Curves.

Column	Depth (cm)	Half-Life (days)		
		Atrazine	Carbofuran	Diuron
A	15	13		
A	60	76	126	194
A	150	35		410
B	15	65		
B	60			339
B	150	33		399
C	15	158	18	
C	30	152	14	274
C	150	138	50	111
D	15	107	19	32
D	30	721	28	266
D	150	238	72	94
E	15	23		
E	30	93	98	132
E	150	20		
F	15	12		
F	30	60	227	292
F	150		5	
H	150		82	

Table 4. Half-Life (days) Estimated from Batch Systems

	Soil		
	Eustis $t_{1/2}$ (CI)	Lincoln $t_{1/2}$ (CI)	Kidman $t_{1/2}$(CI)
Diuron	63 (36 - 230)	95 (41 - 310)	77 (56 - 1700)
Carbofuran	43 (29 - 100)	46 (30 - 110)	40 (29 - 210)
Atrazine	33 (24 - 50)	46 (27 - 130)	58 (37 - 190)

CI = 95% Confidence Interval (days)

Karickhoff (1981, 1984) reported a relationship between the sorption coefficient, solubility, and the melting point of hydrophobic organic chemical sorbates. Thus, it is expected that solubility and melting point are the only variables that could affect the sorption coefficient values for a given hydrophobic organic chemical. If the solubility of hydrophobic organic chemicals is not affected when dissolved in water as a mixture, sorption coefficient values should not be affected either. Hence, competitive sorption of hydrophobic organic chemicals in a multi-sorbate system should not be expected.

Determination of Batch Sorption Isotherms

Equilibrium sorption isotherms for all soil-sorbate combinations were measured at room temperature ($T=24\pm2^{o}C$) using the batch-equilibration method (Nkedi-Kizza et al., 1985a). Soil samples were equilibrated with sorbate solutions in 25 ml amber glass vials with Teflon-lined septa inserts in screw-top caps. Amber vials were used because the phenols undergo photodegradation. Preliminary studies suggested that a 4-hour equilibration time on a platform shaker at low speed was sufficient to attain equilibrium. Both single-sorbate and multi-sorbate systems consisting of binary, ternary, and quinary mixtures of the chemicals were investigated. In all experiments, duplicate or triplicate 5 g of soil samples were equilibrated with 10 ml of electrolyte solution (0.01 N $CaCl_2$) containing a range of sorbate concentrations.

Reversed phase liquid chromatography or liquid scintillation counting methods were used to measure sorbate concentrations in the equilibrium supernatant solutions after centrifuging for 30 minutes at 1200 rev/min with an arm radius of 150 mm (Sorvall RT 6000).

Competitive Sorption

Competitive sorption was determined for the three pesticides. Sorption of these hydrophobic organic chemicals from single-sorbate systems was compared with that in ternary-sorbate systems to evaluate if any competitive sorption occurred. Sorption data were fitted to the Freundlich equation by least-squares optimization methods and compared.

Table 5 contains the results of the single and multiple component isotherms. It is evident that in all cases investigated, the sorption isotherms from single-sorbate and ternary-sorbate systems are essentially identical. This suggests that no competitive effects exist for sorption of these pesticides. The data presented here suggest that sorption from a mixture of hydrophobic sorbates occurs independent of other sorbates in the solution. Although the data are not presented in this paper, the results of this study did indicate that the phenolic compounds exhibited some competitive sorption. This may be related to the partial ionization that phenolics undergo. Perhaps there is a different mechanism of sorption for ionic materials than for neutral hydrophobic organic chemicals.

Conclusions

1. For the pesticides atrazine, diuron, and carbofuran, the presence of other chemicals in the system did not affect their sorption and transport.

2. Apparent degradation rates were observed to vary with time and depth in soil columns. The variation of half-life with time seems to be erratic in nature, indicating fluctuation in microbial population.

Disclaimer

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Table 5. Sorption Coefficients for Single-Sorbate and Ternary Sorbate Systems

		Soil		
	Type	Eustis $K_d \pm CI$	Kidman $K_d \pm CI$	Lincoln $K_d \pm CI$
Diuron	S	1.96 ± 0.23	1.63 ± 0.07	0.75 ± 0.06
Diuron	T	1.76 ± 0.20	1.62 ± 0.05	0.83 ± 0.02
Carbofuran	S	0.06 ± 0.02	0.20 ± 0.03	0.15 ± 0.01
Carbofuran	T	0.06 ± 0.23	0.25 ± 0.01	0.16 ± 0.02
Atrazine	S	0.54 ± 0.06	0.34 ± 0.09	0.20 ± 0.03
Atrazine	T	0.45 ± 0.06	0.30 ± 0.07	0.14 ± 0.02

K_d = Sorption Coefficient (ml/g)
CI = 95% Confidence Interval

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DISCUSSION

SCHOLZ :
J'ai deux questions à vous poser. D'abord, qu'elle était la "philosophie" de l'emploi des eaux usées dans vos expériences ? Ensuite, sur vos sols perturbés, avez-vous mesuré la biomasse en fonction de la profondeur ?
SHORT :
We designed our experiments to simulate the process of land treatment of waste water where the pesticide is incorporated in the waste as a polluant. The experiments were to be used to help decide whether or not pesticides could pollute groundwater at land treatment sites. As for your second question, my response is no. This was not included in the original experiment plan.

MANSOUR :
Vous avez donné des demi-vies pour le carbofuran et l'atrazine ; ces composés ont-ils réellement la même rémanence ?
SHORT :
The values of half-lives in this paper have such a large confidence interval that no conclusion should be made that atrazine and carbofuran degrade at the same rate. Statistically, that would be inappropriate.

DELEU :
Quelles sont les quantités de pesticides appliquées par colonne et sous quelle forme (ponctuelle, continue) ?
SHORT :
We applied approximatively 70 ml and 300 ml of simulated waste water every 6 hours to the low flow and high flow columns respectively.